



FIELD PARAMETER CERTIFICATION GUIDANCE DOCUMENT

Office of Environmental Laboratory Certification

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STANDARD OPERATING PROCEDURES (SOP) MANUAL

Each certified laboratory must have a standard operating procedures (SOP) manual. The SOP must be a detailed manual describing the specific operations of the laboratory for each parameter it is certified to perform, and must be readily available to each analyst. Each method write-up should include, but not be limited to, the following information:

1. Scope and Application
 Type of samples analyzed and analytes of interest.
2. Summary of the Method
 A brief explanation of the procedure.
3. Definitions
 Define terms used in methodology (necessary for new or inexperienced analysts).
4. Interferences
 This would include physical or chemical properties of the sample, contaminants from glassware, reagents, and other apparatus.
5. Safety Apparatus and Procedures
 List any safety items needed to perform the analysis as well as any safety precautions needed.
6. Instrumentation and Equipment
 List all instrumentation and equipment needed to perform the procedure.
7. Reagents and Consumable Materials
 Include manufacturer, catalog number, and any other pertinent information.
8. Sample Preservation and Handling
 List specifics for each program area.
9. Calibration Procedures and Reagent Standardizations
 Specify amounts and concentrations of standards.
10. Approved Analytical Methods and References (EPA or SM)
 This must include all procedures needed for the analysis for the parameters of interest.
11. Quality Control
 Include all laboratory quality control practices.

CHAIN-OF-CUSTODY

Each certified laboratory must have a written procedure for sample handling in its standard operating procedures manual. To ensure sample integrity, it is required that an accurate written record (chain-of-custody) be available to trace the possession and handling of samples from the moment of collection through analysis and final disposition. This is referred to as chain-of-custody and is important in the event of litigation involving the sample results.

Records must include the following:

1. Collection date and time.
2. Sample collector's signature.
3. Unique sample identification number.
4. Sample location and description.
5. Sample type - grab or composite (composite samples must have start/finish time and date for composite sampler).
6. Number of containers.
7. Container type - glass or plastic.
8. Analyses required.
9. Preservatives used - H₂SO₄, NaOH, ice, etc.
10. Program area - WW, DW, GW, etc.
11. Sample matrix - liquid, soil, etc.
12. Transfer signatures w/dates and times for both relinquishment and laboratory receipt (the laboratory should indicate FEDEX, UPS, etc. in the "relinquished to" space if applicable).
13. Receipts maintained when shipped by common carrier (FEDEX, UPS, etc.).

Chain-of-custody forms are not required for field analyses (pH, DO, residual chlorine, and temperature) or any other analyses performed at the collection site (such as specific conductance) since the analyses are performed at the same time that the samples are collected.

SAMPLING PROCEDURES

The objective of sampling is to collect a portion of material sufficient in volume to be transported conveniently and handled in the laboratory while still accurately representing the material being sampled. Valid test results depend on the following:

1. Using proper sampling, sample handling, and preservation techniques.
2. Properly identifying the collected samples and documenting their collection in permanent field records.
3. Maintaining sample chain-of-custody for compliance samples.
4. Protecting the collected samples by properly packing and transporting them to a laboratory for analysis.

Samples must always be collected according to the approach that is specified by the permit.

Grab Samples

A grab sample is a discrete sample collected at a specific point and at a particular instant in time.

Grab sampling is:

1. Used to characterize the material at a particular instant in time.
2. Always associated with instantaneous water and wastewater flow data.
3. Conducted when the water or wastewater stream is not continuous (e.g. batch discharges or intermittent flow).
4. Conducted when the characteristics of the water or waste stream are known to be constant or considered for all practical purposes to be constant.
5. Conducted when the sample is to be analyzed for parameters whose characteristics are likely to change significantly with time, i.e. dissolved gases, bacteria, etc.
6. Conducted when the sample is to be collected for analysis of a parameter such as oil and grease where the compositing process could significantly affect the observed concentration relative to the true concentration.
7. Conducted when data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.

Composite Samples

A composite sample is usually associated with wastewater treatment facility sampling.

A time composite sample is one containing a minimum of eight equal-volume discrete samples taken at equal time intervals over the compositing period. The time composite definition is sometimes used where water or wastewater flows do not vary more than $\pm 15\%$ of the average daily flow rate.

A flow proportional composite sample is one containing a minimum of eight discrete samples with volumes proportional to the flow rate during the compositing period. Flow proportional sampling is usually conducted when the water or wastewater flows vary more than $\pm 15\%$ from the average daily flow rate.

Composite sampling is:

1. Used when average constituent or waste concentrations are of interest.
2. Always associated with continuous flow data.
3. Used when the wastewater stream is continuous.
4. Used when it is necessary to calculate mass per unit time loadings.

Time composite samples will be used where wastewater flows are constant or are considered for all practical purposes to be constant. A time composite sample will be collected continuously or with constant sample volume and a constant time interval between samples.

A flow proportional composite sample is used when wastewater flow is highly variable (greater than $\pm 15\%$ of the average daily flow). A flow proportional composite sample will be collected continuously and proportional to the wastestream flow, with constant sample volume and the time between samples proportional to wastestream flow, or with a constant time interval between samples and a sample volume proportional to flow at the time of sampling.

Continuous Monitoring Devices

Some laboratories may be using continuous monitoring instruments for the measurement of sample pH or residual chlorine. The use of such instruments is approved for compliance monitoring as long as they are operating in accordance with the accepted EPA analysis criteria. Therefore, such instrumentation must be calibrated at least daily to ensure the accuracy of the reported values. Appropriate records of the daily calibrations must be kept to verify that the checks have been performed.

Many of the continuous monitoring instruments now on the market can easily be directly calibrated on a daily basis. Direct calibration of any instrument is always preferred. However, the Department acknowledges that direct daily calibration of some of the continuous monitoring devices currently marketed can be difficult, if not impossible.

As an alternative to direct daily calibration, it will be acceptable for a laboratory to compare a sample value from the continuous monitoring instrument to a sample value obtained from a benchtop instrument that has been properly calibrated each day. For residual chlorine analysis, the value obtained from the continuous monitor must agree within 10% of the value obtained from the benchtop instrument. For pH analysis, the value obtained from the continuous monitor must be within 0.2 pH unit of the value obtained from the benchtop instrument.

PROFICIENCY TESTING (PT) SAMPLES

South Carolina Regulation 61-81 specifies that each laboratory certified by the State of South Carolina which is reporting Clean Water, Safe Drinking Water, or Solid and Hazardous Waste Act data to this Department must report "Acceptable" results for a blind proficiency testing (PT) sample (if available) for each parameter method listed on its environmental laboratory certificate. This requirement must be met to initially be certified and then on an annual basis thereafter.

Once the laboratory is certified, it must successfully analyze a PT sample (obtained from an approved provider) for each parameter and method listed on its environmental laboratory certificate annually.

Water Pollution (WP) and/or Water Supply (WS) Studies, as applicable, must be performed each year in order to maintain certification for the time period specified on the certification certificate. It is important that the laboratory participate in these studies as early as possible to avoid potential loss of certification. The studies must be part of an official WP or WS study, begin and end within the calendar year, and be graded and reported by the PT Provider to this Office no later than December 31 of each calendar year. Results received after December 31 cannot be used for compliance with the PT requirement. Split studies may be used for recertifying a laboratory but may not be used to meet the annual PT requirement. Split studies are those studies that open in one year and are graded and reported the following year or studies that open in one year and close in another year. If the laboratory has questions regarding the submission of PT data, the acceptability of specific studies, or has questions about the annual requirement, please contact this Office.

Please also note that if you fax, e-mail, or enter your results on-line, we strongly recommend that you also mail them via postal mail or contact the Provider to ensure that the e-mail, internet data entry, or fax was received in its entirety. There have been instances where the PT Provider did not receive the results and the laboratory was decertified.

TEMPERATURE

Temperature measurements can be made with glass/mercury, glass/alcohol or dial bimetallic Celsius (°C) thermometers (graduated in 0.1, 0.2, 0.5, or 1°C increments). The devices must be checked for accuracy using a NIST or NIST-traceable reference thermometer. Glass/mercury and glass/alcohol thermometers must be checked at least annually and dial bimetallic thermometers must be checked at least quarterly.

Any field meters equipped with temperature sensors are used to make temperature measurements for compliance purposes, then they must also be checked for accuracy against a NIST or NIST-traceable reference thermometer at least annually.

Thermometer Accuracy Check Procedure

1. Immerse laboratory thermometer (or other temperature sensing device) into a sample at the normal range of use for the thermometer or device.
2. Immerse the reference thermometer into the sample.
3. Allow both thermometers to stabilize.
4. Record the readings from both thermometers and note the difference.
5. Document thermometer accuracy checks with the following information:
 - a) NIST reference thermometer I.D. #.
 - b) laboratory thermometer I.D. #.
 - c) date of accuracy check.
 - d) NIST reference thermometer reading.
 - e) laboratory thermometer reading.
 - f) temperature correction.
 - g) analyst's initials.
6. Each thermometer, upon being checked for accuracy must be tagged with the following information:
 - a) date of accuracy check.
 - b) initial's of the analyst performing the accuracy check.
 - c) temperature difference from the reference thermometer - which must be taken into account each time a temperature measurement is made in order to get an accurate reading.

Sample Analysis Procedure

1. Collect sample and analyze immediately (as soon as possible, not to exceed 15 minutes).
2. Immerse thermometer in sample.
3. Allow thermometer to stabilize.
4. Note temperature reading and compensate for any difference with the reference thermometer.
5. Record corrected temperature reading.

TEMPERATURE CHECKLIST

1. Type of thermometers/temperature sensors used:
 - a)
 - b)
 - c)
2. Source of NIST/NIST-traceable reference thermometer:
3. Is certificate for reference thermometer available?
4. Accuracy check records for thermometers and temperature sensors used?

Records include:

 - a) date.
 - b) reference thermometer number.
 - c) reading of reference thermometer.
 - d) identification of thermometer checked.
 - e) reading of thermometer checked.
 - f) difference/adjustment.
 - g) comments.
 - h) initials.
5. Thermometers/temperature sensors labeled with
 - a) date checked.
 - b) analyst.
 - c) correction value/adjustments.
6. Correction value/adjustment taken into account?

HYDROGEN-ION CONCENTRATION (pH)

Equipment and Supplies

1. A pH meter capable of reading pH values of a solution ranging from 0 - 14 standard units (SU), in 0.1 SU increments, with the appropriate electrode. The meter's accuracy must be no less than ± 0.1 SU.
2. Reference buffers of pH 7.0 and 4.0 or 10.0.
3. Deionized or distilled water, preferably in a squeeze bottle.
4. Small beakers (glass or teflon) for holding buffer solutions during calibrations.
5. Laboratory benchsheet for documentation of calibration and analysis.
6. Optional:
 - a) magnetic stirring bar.
 - b) magnetic stirring plate - please be aware that the stirring plate can generate heat over an extended period of time and change the temperature of buffer solutions and samples.
 - c) lint free tissues, such as Kim-Wipes - as recommended by manufacturer

Calibration Criteria

1. The instrument must be calibrated at two points using two different buffers. The pH 7.0 buffer must be used and the laboratory must select either a pH 4.0 or pH 10.0 buffer for the second point. The two buffers chosen must bracket the expected pH value of the sample to be analyzed as closely as possible. For example, if the analyst expects the pH of the sample to be 7.9, then pH 7.0 and 10.0 buffers should be used for the calibration.
2. It is recommended that a third buffer be used to check the accuracy of the meter. A three-point calibration can be performed.
3. Each shift the meter must be recalibrated.
4. For facilities that have to perform field analysis at multiple sites, the pH meter can be initially calibrated in the laboratory and then checked with a pH 7.0 buffer at each site before sample analysis.
5. Buffers must not be used more than once. At each calibration, a fresh aliquot of buffer must be used.
6. Documentation of all calibrations must be maintained and must be readily available for examination. The records must, at a minimum, include the following information:
 - a) date of calibration.
 - b) time of calibration.
 - c) initials of analyst.
 - d) meter I.D. (if more than one meter is used by the laboratory).
 - e) reading for the pH 7.0 buffer after meter adjustment.
 - f) reading for the pH 4.0 or 10.0 buffer after meter adjustment.
 - g) slope and/or efficiency if given by the meter (the slope/efficiency reading is used as an indicator of electrode problems)
 - h) comment section.

7. Buffers must not be used after their expiration dates, as provided on the container by the manufacturer. If the manufacturer fails to provide an expiration date, the laboratory must assign an expiration date of one year after opening in the laboratory. The date received in the laboratory, the date opened, and the expiration date must be recorded on all containers of pH buffer.
8. When buffers are transferred to a secondary container for temporary storage, the lot #, buffer pH value, expiration date, and analyst's initials must be written on the container.
9. Buffer solutions prepared by the laboratory from pH powder pillows or liquid concentrates can be used up to one year from the date that they are prepared. The container must be labelled with the date prepared, lot # of the pillows, buffer pH value, expiration date, and preparer's initials.
10. The instrument must be calibrated each day before it is used. When the tip of the electrode is submerged in the buffer during calibrations, gently swirl it until the meter reading stabilizes. Alternatively, a magnetic stirring bar and stirrer can be used.
11. When transferring the electrode from one buffer to another, the electrode must be rinsed with deionized or distilled water and gently wiped or blotted with a lint-free tissue (unless discouraged by the manufacturer).
12. The electrode must be stored and maintained according to the manufacturer's instructions. This information must be included in the SOP.
13. Document any maintenance performed on the meter or the electrode. Meter must be recalibrated before use after any maintenance.
14. If the instrument is equipped with an automatic temperature compensator (ATC), it is recommended that the device be checked for accuracy with a NIST or NIST-traceable reference thermometer. See TEMPERATURE section of this document for details. If the instrument is used for reporting temperature, the sensor must be checked annually.

Analysis Criteria

1. The pH meter must be calibrated before any analyses are performed.
2. The analyst must maintain documentation of all analyses. The records must include, at a minimum, the following information:
 - a) date of analysis
 - b) time of sample collection and time of sample analysis
 - c) analyst's initials
 - d) instrument ID (if more than one meter is used by the laboratory)
 - e) sample identification
 - f) pH value of sample
 - g) comment/maintenance section
3. If more than one sample is analyzed in a series, the electrode must be rinsed with deionized or distilled water and blotted gently with a lint-free tissue (unless discouraged by the manufacturer) between samples.
4. When analysis is complete, the electrode must be rinsed and stored according to the manufacturer's instructions.
5. Instrument maintenance must be performed as required by the manufacturer.

HYDROGEN-ION CONCENTRATION (pH) CHECKLIST

1. Manufacturer:
2. Instrument and electrode clean?
3. Buffers within expiration dates? 7 ___ 4 ___ 10 ___
4. Electrode:
 - a) storage solution.
 - c) if nonsealed, filled to proper level with electrolyte solution?
5. Buffer aliquots changed daily/used only once?
6. Deionized/distilled water in squeeze bottle, used to rinse electrode between samples and buffers?
7. If buffers transferred to secondary containers for use and storage, pH value, expiration date, analyst's initials, and lot number written on secondary containers?
8. Instrument calibrated before use?

Calibration records must include:

 - a) date
 - b) time
 - c) analyst
 - d) instrument identification (if more than one instrument is used)
 - e) Documentation of the pH 7 buffer checks
 - f) Documentation of the pH 4 and/or 10 buffer checks*
*Buffers must bracket sample values.
 - g) third buffer check (optional - highly recommended)
 - h) slope or efficiency (if given by instrument)
 - i) comments/maintenance
9. Sample analysis records must include:
 - a) date
 - b) time sample is collected
 - c) time sample is analyzed
 - d) analyst's initials
 - e) instrument ID (if more than one instrument is used)
 - f) sample identification
 - g) pH value
 - h) comments/maintenance
10. Is sample analyzed within 15 minutes of collection?
11. Is sample stirred gently during analysis?

DISSOLVED OXYGEN (DO) – Membrane Electrode Method

Equipment and Supplies

1. An oxygen-sensitive membrane probe and the appropriate meter
2. Laboratory benchsheet for documentation of calibration and analysis

Calibration Criteria

1. The instrument must be calibrated each day before it is used.
2. The instrument must be calibrated according to the manufacturer's instructions.
3. Documentation of all calibrations must be maintained by the laboratory and must be readily available for review. Calibration records must reflect the calibration procedure required by the manufacturer. The records must, at a minimum, include the following information provided it is applicable to the instrument in use:
 - a) date and time of calibration.
 - b) meter ID, if more than one meter is used by the laboratory
 - c) analyst's initials
 - d) membrane check
 - e) comment/maintenance section
 - f) adjusted/final DO value

Depending on the type of meter in use, the following information must be recorded. If the laboratory's meter provides or requires the following, it must be recorded. If the meter does not provide or require the following, the information is not required.

- g) air temperature
 - h) initial DO value
 - i) zero check
 - j) red-line check
 - k) altitude adjustment
 - l) battery check
4. The DO probe must be stored according to the manufacturer's instructions
5. The DO probe's temperature sensor must be checked for accuracy at least once a year using a NIST or NIST-traceable reference thermometer. This is required even if the meter is not used for reporting temperature. This check must be recorded on a thermometer check log. The probe must then be tagged with the following information:
 - a) date of temperature check
 - b) initials of analyst performing check
 - c) temperature difference between the probe and the NIST-traceable thermometer
6. If the temperature difference from the reference thermometer is more than 1.0°C, then the probe must be repaired or replaced.
7. Records of maintenance performed on the instrument must be kept for review (e.g. membrane, battery, and probe changes). This information can be placed in the comments section of the sample analysis record.

Analysis Records

1. The instrument must be calibrated before any sample analyses can be performed.
2. The analysis records must include, at a minimum, the following information:
 - a) date of analysis
 - b) time of sample collection and sample analysis
 - c) sample I.D. (name or number)
 - d) dissolved oxygen (DO) reading in mg/L (ppm)
 - e) analyst's initials
 - f) comments
 - g) instrument ID if more than one instrument is used in the laboratory

DISSOLVED OXYGEN (DO) CONCENTRATION CHECKLIST

1. Manufacturer:
2. Instrument and probe clean?
3. Membrane in good condition?
Wrinkles, tears, air bubbles?
4. How is probe stored?

When not in use -
During saturated air calibration -
5. Temperature sensor checked for accuracy?
6. Instrument calibrated before use?
Calibration records include:
 - a) date and time of calibration
 - b) meter number if more than one meter is used by the laboratory
 - c) analyst's initials
 - d) membrane check
 - e) comment/maintenance section
 - f) adjusted DO value

Depending on the type of meter in use, the following information must be recorded. If the laboratory's meter provides or requires the following, it must be recorded. If the meter does not provide or require the following, the information is not required.

 - g) air temperature
 - h) initial DO value
 - i) zero check
 - j) red-line check
 - k) altitude adjustment
 - l) battery check
7. Sample analysis records must include:
 - a) date of analysis.
 - b) time of sample collection and sample analysis
 - c) sample I.D. (name or number)
 - d) dissolved oxygen (DO) reading in mg/L (ppm)
 - e) analyst's initials
 - f) comments
 - g) Instrument ID, if more than one instrument is used in the laboratory
8. Is sample analyzed within 15 minutes?
9. Is sample stirred gently during analysis?

RESIDUAL CHLORINE – DPD Spectrophotometric or Colorimetric

Equipment and Supplies

1. A spectrophotometer or colorimeter, for use at a wavelength of 515 nm and providing a light path of 1 cm or longer
2. Spectrophotometer cells (vials)
3. DPD reagent powder pillows
4. Volumetric glassware to prepare standards
 - a) volumetric pipets
 - b) volumetric flasks
5. Potassium permanganate (KMnO_4) or commercially prepared chlorine stock standard solutions may be used
 - a) Potassium permanganate (KMnO_4) primary stock solution (1000ppm) - 0.891g KMnO_4/L
 - 1) made fresh at least every 6 months
 - 2) stored in refrigerator
 - 3) stored in a glass container protected from light
 - 4) labelled with date prepared, reagent name and concentration, and analyst's initials
 - b) Commercially prepared KMnO_4 primary stock solution (1000ppm) - 0.891g KMnO_4/L
 - 1) purchased in an amber glass container
 - 2) unopened container good up to the manufacturer's expiration date
 - 3) shelf-life of six months once opened*
*The six month shelf-life will take precedence over any extended expiration date that the manufacturer may have assigned but cannot exceed the manufacturer's expiration date, if provided.
 - 4) stored in refrigerator
 - 5) once opened, labeled with date opened, six month expiration date, and analyst's initials
 - c) Secondary stock solution (100ppm) - dilute 10 ml of primary stock to 100 ml
 - 1) made fresh at least every week
 - 2) stored in refrigerator
 - 3) stored in a glass container protected from light
 - d) Working standards
 - 1) made fresh daily in glass containers
 - 2) a minimum of two concentrations used daily
- If degradation is observed or standard readings differ more than 10% of the standard values, new secondary and even primary standard solutions must be made before the normal expiration dates.
6. Commercially prepared liquid chlorine standard solution must be used according to the manufacturer's instructions and diluted to prepare working standards. Gel standards are not approved for use.

- a) Working standards
 - 1) standards made fresh daily
 - 2) a minimum of two concentrations used daily

Initial Calibration Verification

1. An initial calibration verification (using 5 standards) must be performed annually or when a new lot number of DPD reagent pillows is used, whichever is sooner.
2. A separate initial calibration verification must be generated for DPD free and total reagent powder pillows.
 - a) a separate calibration verification must be generated for each type of sample vial (2.5 cm and 1.0 cm, other) used
3. A minimum of 5 standards and a blank covering the range of the instrument's capabilities must be used to generate each calibration verification.
4. Each initial calibration verification must, at a minimum, be labeled with the following information:
 - a) date and time generated
 - b) analyst's initials
 - c) instrument I.D. number (if more than one instrument is used by the laboratory)
 - d) lot number of DPD reagent pillows
 - e) cell path length (if interchangeable)
 - f) true concentration of standards
 - g) observed concentration (meter readings) or absorbance of standards
 - h) wavelength (if adjustable)
 - i) record of true standard concentration vs. observed standard concentrations. All observed (meter readings) must be within 10% of the true standard concentration. If not, prepare fresh standards. If the 10% criteria still cannot be met, send meter for service.
 - j) identification of results as total or free residual chlorine

Daily Calibration Verification Criteria

1. The instrument calibration must be verified daily before it is used
2. A minimum of two standards and a blank must be used for daily calibration verification. The observed concentration for each standard must be within 10% of its true concentration.
3. The daily calibration verification records must be readily available for examination and must, at a minimum, include the following information:
 - a) date of calibration
 - b) time of calibration
 - c) instrument I.D. number if more than one is used in the laboratory
 - d) blank analysis
 - e) true concentration of standards
 - f) observed concentration for each standard. The observed value must be within 10% of the true value.
 - g) lot number of DPD reagent pillows
 - h) initials of analyst
 - i) wavelength (if adjustable)

- j) cell path length (if interchangeable)
- k) identification of results as total or free chlorine

Analysis Criteria

1. The instrument's calibration must be verified each shift before conducting sample analysis.
2. Documentation of all analyses must be readily available for examination and must, at a minimum, include the following information:
 - a) date of analysis
 - b) time of analysis (samples must be analyzed within 15 minutes after collection)
 - c) analyst's initials
 - d) instrument used (if more than one is used in the laboratory)
 - e) sample identification
 - f) sample chlorine value, mg/l (ppm)
 - g) lot number of DPD reagent pillows. (Must be traceable to the initial and daily calibration verification.)
 - h) comment section
 - i) Identification of results as total or free chlorine
3. When reporting results, the laboratory cannot report values that are lower than the lowest standard concentration used to generate the initial calibration verifications. For example, if the lowest standard used to perform the initial calibration verification is 0.1 mg/L, the minimum reporting limit is 0.1 mg/L. If the meter reads less than 0.1 mg/L, then the analyst should report the value as "<0.1 mg/L". Daily calibration verifications can be performed with higher or different standard concentrations. For example, 0.5 and 1.0 mg/L standards can be used for daily calibrations.

TOTAL RESIDUAL CHLORINE CHECKLIST

1. Instrument:
Instrument and equipment (vials) clean:
2. SOP manual
3. Initial Calibration Verification
 - a) Generated annually or when lab uses a new lot of DPD pillows
 - b) Use 5 standards and blank covering range of meter capabilities
 - c) Labeled with:
 - 1) date generated and time generated
 - 2) analyst's initials
 - 3) instrument I.D. (if more than one instrument is used in the laboratory)
 - 4) lot number of DPD reagent pillows
 - 5) cell path length (if adjustable)
 - 6) true concentration of standards
 - 7) observed concentration (meter reading) of standards
 - 8) wavelength (if adjustable)
 - 9) record true concentration vs. observed (meter reading) concentration . All observed (meter readings) must be within 10% of the true standard concentration. If not, prepare fresh standards. If the 10% criteria still cannot be met, send meter for service.
 - 10) identification as total or free chlorine
4. Stock solutions and standards
 - a) Primary stock solution (1000ppm) - 0.891g/L potassium permanganate
 - 1) made fresh at least every 6 months
 - 2) stored in refrigerator
 - 3) stored in a glass container protected from light
 - 4) labeled with date prepared and initials of analyst
 - b) Commercially prepared KMnO_4 primary stock solution (1000ppm) - 0.891g KMnO_4 /L
 - 1) purchased in an amber glass container
 - 2) unopened container within the manufacturer's expiration date
 - 3) opened containers within 6 month holding time*
 - 4) stored in refrigerator in a glass container
 - 5) once opened: labeled with date opened, six month expiration date, and analyst's initials

*The six month shelf-life will take precedence over any extended expiration date that the manufacturer may have assigned but may not exceed the manufacturer's expiration date.
 - c) Secondary stock solution (100ppm)
 - 1) 10 ml of 1000ppm stock to 100 ml
 - 2) made fresh weekly
 - 3) stored in a glass container in a refrigerator
 - 4) stored protected from light

- d) Commercially Prepared Chlorine Standards
 - 1) used according to manufacturer's instructions
 - 2) diluted to prepare working standards
- e) Calibration Verification Working Standards
 - 1) Initial Calibration Verification - five concentrations covering the range of the analyses
 - 2) Daily Calibration Verification - two concentrations in working range
 - 3) Both initial and daily calibration working standards made fresh daily
- f) Volumetric glassware used to prepare standards
 - 1) volumetric pipets
 - 2) volumetric flasks
- g) Daily Calibration Verification
 - 1) Calibration verification performed daily, before use of instrument
 - 2) Use of two standards and a blank
 - 3) Calibration records include:
 - a) date of calibration
 - b) time of calibration
 - c) instrument I.D. (if more than one instrument is used by the laboratory)
 - d) lot number of DPD reagent pillows (must be traceable to the initial calibration)
 - e) true concentration of standards
 - f) observed (meter reading) concentration of standards. (must be within 10% of the true value)
 - g) analyst's initials
 - h) identification of results as total or free chlorine
- h) Analysis records include:
 - 1) date
 - 2) time sample collected and analyzed
 - 3) instrument I.D. (if more than one instrument is used by the laboratory)
 - 4) sample reading, mg/L (ppm)
 - 5) analyst's initials
 - 6) lot number of DPD reagent pillows (must be traceable to the initial calibration and daily calibration)
 - 7) identification of results as total or free chlorine
- j) Maintenance records
 - 1) batteries replaced.
 - 2) service documented.

REFERENCES

1. Standard Methods for the Examination of Water and Wastewater, 18th, 19th and 20th Edition.
American Public Health Association.
2. 40 CFR Parts 141 and 142 for Safe Drinking Water Act.
3. 40 CFR Part 136 for Clean Water Act.

THERMOMETER CHECK RECORD

- A. National Institute of Standards and Technology (NIST) Certified Thermometer or equivalent thermometer identification number: _____
- B. Identification number of thermometer that is being calibrated, identified as "B".
Thermometers must be checked in the range of use. For example: If the thermometer is used in the refrigerator to monitor 0-4°C, the thermometer must be checked against the NIST-traceable thermometer at 4°C.

[illegible]